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Giant birefringence and dichroism induced by ultrafast laser pulses in hydrogenated amorphous silicon

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A femto- and picosecond laser assisted periodic nanostructuring of hydrogenated amorphous silicon (a-Si:H) is demonstrated. The grating structure with the subwavelength modulation of refractive index shows form birefringence ($\Delta n \approx -0.6$) which is two orders of magnitude higher than commonly observed in uniaxial crystals and femtosecond laser nanostructured silica glass. The laser-induced giant birefringence and dichroism in a-Si:H film introduce extra dimensions to the polarization sensitive laser writing with applications that include data storage, security marking, and flat optics. © 2015 AIP Publishing LLC. [<http://dx.doi.org/10.1063/1.4919538>]

Form birefringence, which results from spatially inhomogeneous patterns, may exceed the natural birefringence of crystals by several orders of magnitude.¹ The giant birefringence has been developed by the means of chemical material processing or nanolithography techniques in various systems including optical antenna arrays,² surface nanogratings,^{3–5} ensembles of nanowires,^{6,7} photonic crystals,⁸ and nanophotonic waveguides.⁹ The opportunity to design optically anisotropic surfaces is of special interest because it opens a way to control the light wave front by altering its polarization creating in particular, the geometrical Pancharatnam-Berry phase.¹⁰ Theoretically any phase pattern can be achieved solely by means of the geometrical phase with the efficiency reaching 100%.¹¹ One of the promising techniques for implementing optical elements with a geometrical phase is femtosecond laser assisted nanostructuring.¹² It has been demonstrated that materials irradiated with femtosecond laser pulses undergo the self-assembly process, which results in nanostructures produced on the surface^{13,14} or inside the material.^{15,16} Recently, this technology was harnessed for producing a series of birefringent optical elements inside silica glass,^{12,17–19} while the optical anisotropy produced by laser-induced periodic surface structures (LIPSS) has not been reported. However, as the form birefringence depends on the refractive index modulation,¹ the amount of form birefringence achieved in nanostructured silica glass²⁰ is of the order of natural birefringence found in crystalline materials such as quartz. In order to enhance the optical anisotropy, a material with a high refractive index is required. A promising candidate is hydrogenated amorphous silicon (a-Si:H), which plays a significant role in the world's production of electronics and is ubiquitous for many applications ranging from liquid crystal displays to medical sensors and solar

cells.²¹ Transforming amorphous silicon (a-Si) into a nanocrystalline form through ultrafast laser irradiation has already been demonstrated (nc-Si).^{22–31} In this letter, we demonstrate that the ultrafast laser assisted nanocrystallization of a-Si:H is accompanied by the formation of a laser-induced periodic structure. The observed pattern with the subwavelength modulation of the complex refractive index exhibits a dichroism and enhanced form birefringence, which is two orders of magnitude higher than commonly observed in uniaxial crystals such as quartz, ruby, sapphire, or femtosecond laser nanostructured silica glass. The proposed technology allows implementing functional optical elements with a sub-micron thickness by exploiting the laser imprinted geometrical phase patterns. Both birefringence and dichroism can be controlled by changing laser writing parameters adding extra dimensions to polarization sensitive printing, which is technologically attractive for flat optics,¹¹ optical data storage,³² and security marking.

Experiments were carried out with 300 nm thick a-Si:H films deposited on silica glass by the plasma-enhanced chemical vapour deposition (PECVD) upon the decomposition of the mixture of silane (SiH₄) and argon (Ar) at substrate temperature of 250 °C. The ratio between the gases in the CVD chamber was 25% SiH₄ and 75% Ar.

In the experiments, the a-Si:H films were irradiated with the mode-locked Yb:KGW ultrafast laser system (Pharos, Light Conversion Ltd.) operating at $\lambda_L = 1030$ nm (photon energy ~ 1.2 eV) and delivering pulses with a 100 kHz repetition rate. The pulse duration ranging from 360 fs to 4.8 ps with the pulse energy varying from 0.005 μ J to 0.2 μ J was employed. The laser beam, polarized parallel or perpendicular to the writing direction, was focused onto the film surface via a 0.13 NA-objective lens producing the fluence of 0.0068–0.27 J/cm², or intensity of 1.89×10^{10} – 7.5×10^{11} W/cm² for 360 fs and 1.42×10^9 – 5.6×10^{10} W/cm² for 4.8 ps, respectively. The estimated spot size of the laser beam in the

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focus was $\omega_0 \approx 5 \mu\text{m}$. For all parameters (polarization, pulse duration, and laser energy), $80 \mu\text{m}$ long lines were written with an interline distance of $30 \mu\text{m}$ at a 0.2 mm/s translation speed.

Irradiated regions were analysed with a scanning electron microscope (SEM) (Zeiss Evo50) and an optical microscope (Olympus BX51). Raman spectroscopy was performed with a Renishaw inVia system operating at 632.8 nm excitation. The system was calibrated according to the mono-crystalline silicon (Si) line at 520.7 cm^{-1} . To avoid heat induced crystallization, the excitation power was restricted to 0.1 mW .

Structural and optical analysis of irradiated regions revealed several modification regimes (Fig. 1). “Type 0” modification occurs at the energies of $0.025\text{--}0.03 \mu\text{J}$ and is distinguished only for the pulse duration shorter than 2 ps . Optically, this modification manifests as a darkening of the film. According to Zanzucchi *et al.*,³³ “Type 0” regime appears due to enhanced optical absorption in visible spectrum and is attributed to the hydrogen (H) release. The Raman analysis of the wagging vibrations of Si-H studied elsewhere³¹ confirms that the H release starts at the energies below $0.02 \mu\text{J}$, and at $0.03 \mu\text{J}$ its content drops to 20% of the initial value.

For “Type I” regime, at the energies slightly below $0.07 \mu\text{J}$ for 360 fs or $0.05 \mu\text{J}$ for 4.8 ps , amorphous silicon with the broad Raman band at 480 cm^{-1} begins to be replaced by the crystalline silicon transverse optical (TO) phonon mode with the peak position at $518.0\text{--}519.2 \text{ cm}^{-1}$, which we attribute to the formation of nc-Si (Fig. 2). Using the confinement model,³⁴ the silicon grain size of $4\text{--}6 \text{ nm}$ was estimated. The morphology analysis revealed that the nc-Si grains are aggregated into sub- 100 nm domains, which are elongated parallel to the beam polarization and organised into high spatial frequency structure with the periodicity defined by the domain width ($\Lambda \approx \lambda_L/10$) (Fig. 3(top)).

Both the energy and pulse duration affect the position and full width at half maximum (FWHM) of nc-Si TO mode (see Fig. 2(b)). The upshift in frequency and the narrowing of the band indicates the formation of larger silicon grains up to $8\text{--}9 \text{ nm}$ size at higher pulse energies in the case of 360 fs

laser pulses. In contrast, the treatment with 4.8 ps pulses always results in development of smaller crystallites reaching $\sim 5 \text{ nm}$ size.

At high energies ($>0.09 \mu\text{J}$ for 360 fs , and $>0.06 \mu\text{J}$ for 4.8 ps) slightly above the ablation threshold, the film starts to crystallize along its depth (“Type II”), periodically organizing into a nanograting oriented perpendicular to the laser beam polarization (Figs. 1–3). The average period of the structure is $\Lambda \approx \lambda_L/3$ that corresponds to $\lambda_L/n_{\text{film}}$. Such a subwavelength modulation of high-contrast refractive index ($n_{\text{air}}/n_{\text{film}}$) originates from birefringence.¹ Simultaneously a non-zero imaginary part of the material’s complex refractive index (extinction k) leads to the polarization sensitive absorption coefficient ($\alpha = 4\pi k/\lambda$), i.e., dichroism.

The laser-induced birefringence and dichroism were characterized with the quantitative birefringence measurement system (CRi Abrio; Olympus BX51) operating at $\lambda = 546 \text{ nm}$. In this system, the dichroism manifests itself as an artificial birefringence added to the system.³⁵ To recover the absolute values of the birefringence and dichroism, the measured data were additionally processed with a mathematical algorithm.

Figures 4(a) and 4(b) show the retardance and dichroism extracted from the set of 1600 lines imprinted in a-Si:H film. With respect to threshold energy diagram (Fig. 1), the enhanced birefringence corresponds to “Type II” regime, while “Type I” exhibits roughly two orders of magnitude lower birefringence values (Fig. 4(a)). Upon 360 fs , the threshold values of $0.045 \mu\text{J}$ for “Type I” regime and $0.1 \mu\text{J}$ for “Type II” regime were identified. While for 4.8 ps , these thresholds are reduced to $0.035 \mu\text{J}$ and $0.065 \mu\text{J}$, respectively. The laser-induced dichroism follows the “Type II” modification (Fig. 4(b)).

According to the Raman spectra shown in Fig. 2, at the pulse energies higher than $0.1 \mu\text{J}$, irradiation with femtosecond pulses results in larger grains and is 2–3 times more efficient than that with picosecond pulses. Such a pulse duration dependent lattice transformation indicates that the light intensity plays the dominant role, i.e., the lattice transformation starts within the first pulses, when only a-Si:H is present. Specifically, at the initial stage of the irradiation, the intense femtosecond pulses generate more defects (in comparison with picosecond ones) by reducing the hydrogen content in the lattice. Assuming that most of the hydrogen is released before the crystallization and laser-induced nanostructuring,³¹ the absorption of the film increases drastically. This is because the absorption coefficient of the produced a-Si (at photon energy of $\sim 1.2 \text{ eV}$) is almost two orders of magnitude higher than the nc-Si and four orders higher than the a-Si:H,^{32,35–38} i.e., a-Si defines the efficiency of the subsequent modification regimes. Over the further multi-pulse irradiation, the absorption dependent nc-Si content increase leads to the a-Si:H/a-Si content decrease. As a result, the threshold energy of enhanced retardance and dichroism due to higher absorption starts decreasing with the increase of pulse duration ($\tau > 1 \text{ ps}$) (Figs. 4(a) and 4(b)).

Although “Type II” threshold energy depends on the pulse duration, the peak values of retardance $R \approx 150\text{--}180 \text{ nm}$ (phase shift of $\sim 0.7\pi$, at $\lambda = 546 \text{ nm}$) and dichroism $D \approx 0.6\text{--}1$ remain roughly the same ranging from 360 fs to 4.8 ps at

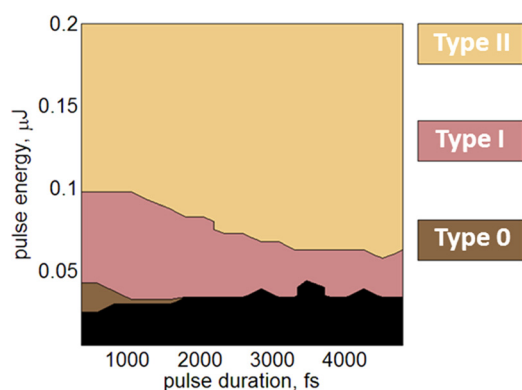


FIG. 1. The threshold energies for a-Si:H modification regimes: Type 0—H release; Type I—silicon crystallization followed by the high spatial frequency structure with low birefringence; Type II—silicon crystallization followed by the nanograting structure that corresponds to the laser-induced dichroism and giant birefringence. Black region indicates the conditions when the change in film structure was not observed.

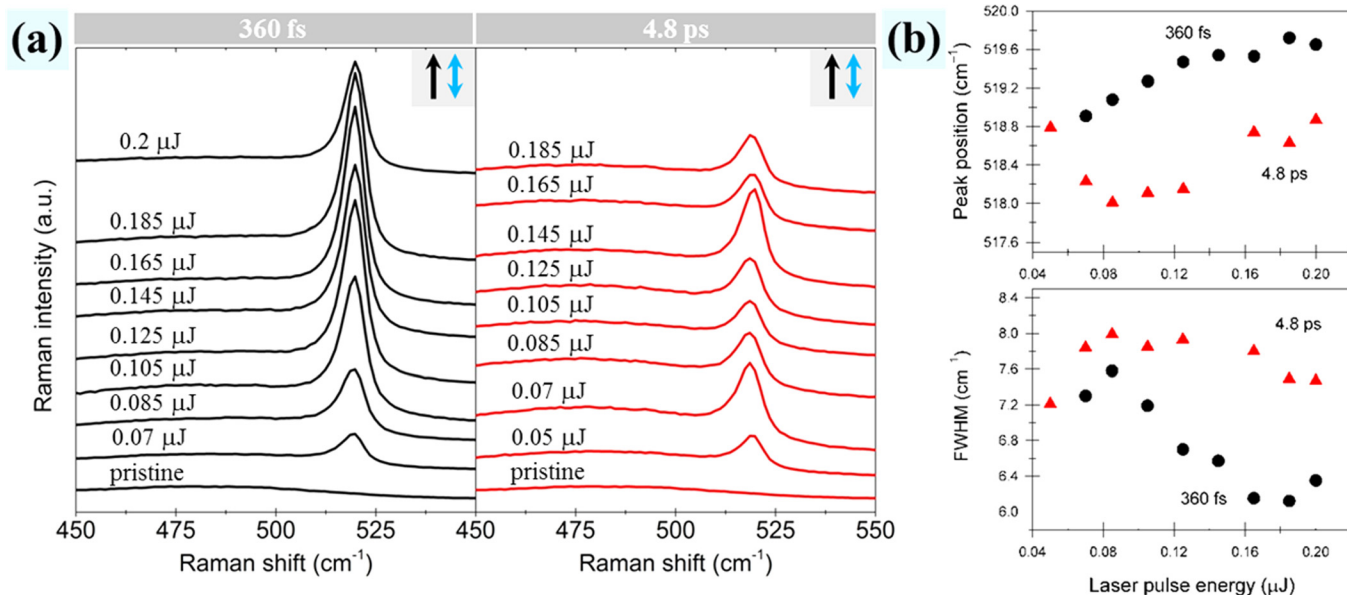


FIG. 2. (a) Raman spectra of a-Si:H treated by 360 fs (black) and 4.8 ps (red) laser pulses with the energy of 0.05–0.2 μJ . The system was operating at 100 kHz with writing speed of 0.2 mm/s. The bottom curves show the Raman spectra of untreated a-Si:H film. Black arrows indicate the laser writing direction, blue arrows—the state of polarization. (b) Effect of laser pulse energy and pulse duration on the peak position (upper panel) and full width at half maximum (FWHM) (bottom panel) of TO mode of produced nc-Si grains.

pulse energies lower than 0.15 μJ (Figs. 4(a) and 4(b)). The retardance is defined as

$$R(\lambda) = d \times (n_e(\lambda) - n_o(\lambda)) = d \times \Delta n(\lambda), \quad (1)$$

where d is the thickness of the film and n_e, n_o —extraordinary and ordinary effective refractive indices. Thus the birefringence of nanograting with $d = 300 \text{ nm}$ and corresponding retardance values is estimated as $\Delta n \approx -0.5$ to -0.6 . The birefringence induced by ultrafast laser nanostructuring is comparable to the giant birefringence achieved in amorphous silicon by electron-beam nanolithography.³ For comparison, the typical values of $n_e - n_o$ imprinted by the femtosecond laser writing inside the silica glass are roughly -5×10^{-3} .²⁰ However, when the pulse energy is higher than 0.15 μJ , the nc-Si content decreases (Fig. 2(a)). The “Type II” regime is

accompanied by the silicon oxidation, apparent due to the enhanced oxygen incorporation into the silicon lattice at high pulse energies.^{28,39} As a result, form birefringence is reduced to -0.4 due to the refractive index change from crystalline silicon to silica glass (Fig. 4(a)).

The dispersion of retardance and dichroism was investigated by using a VIS-NIR birefringence measurement system (CRAIC; Olympus BX51). The transmittance of light was measured when the polarizer before the sample was fixed at 45° with respect to the nanograting orientation while the polarizer after the sample was fixed at 45° (I_{parallel}) or -45° (I_{crossed}). The retardance dispersion $R(\lambda)$ was then calculated as follows:⁴⁰

$$R(\lambda) = \arccos\left(\frac{I_{\text{parallel}} - I_{\text{crossed}}}{I_{\text{parallel}} + I_{\text{crossed}}}\right) \times \frac{\lambda}{2\pi} = \Delta(\lambda) \times \frac{\lambda}{2\pi}. \quad (2)$$

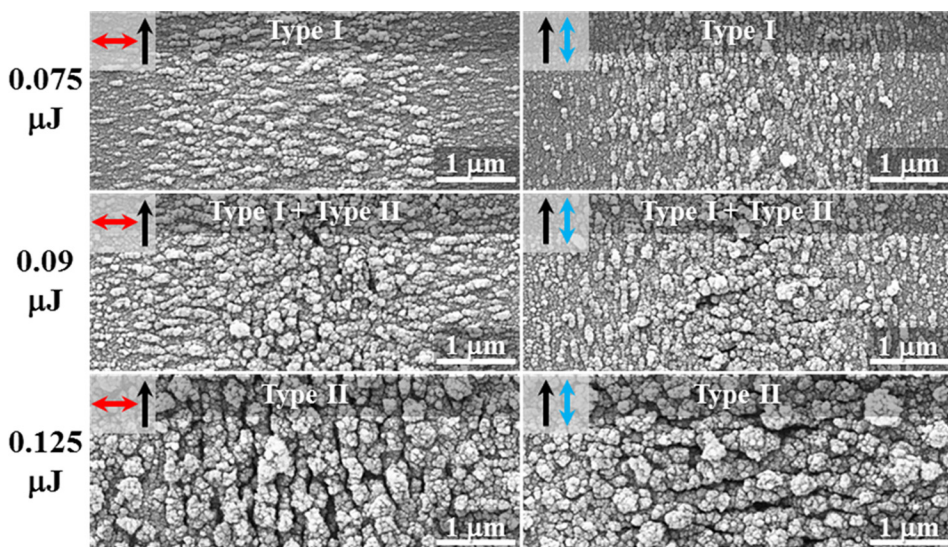


FIG. 3. SEM images of the laser modified a-Si:H film. The shown single lines were written with 0.075 μJ , 0.09 μJ , 0.125 μJ pulse energy. The system was fixed to 100 kHz, 360 fs pulse duration and 0.2 mm/s writing speed. Black arrows indicate the laser writing direction, while red/blue arrows—the polarization state.

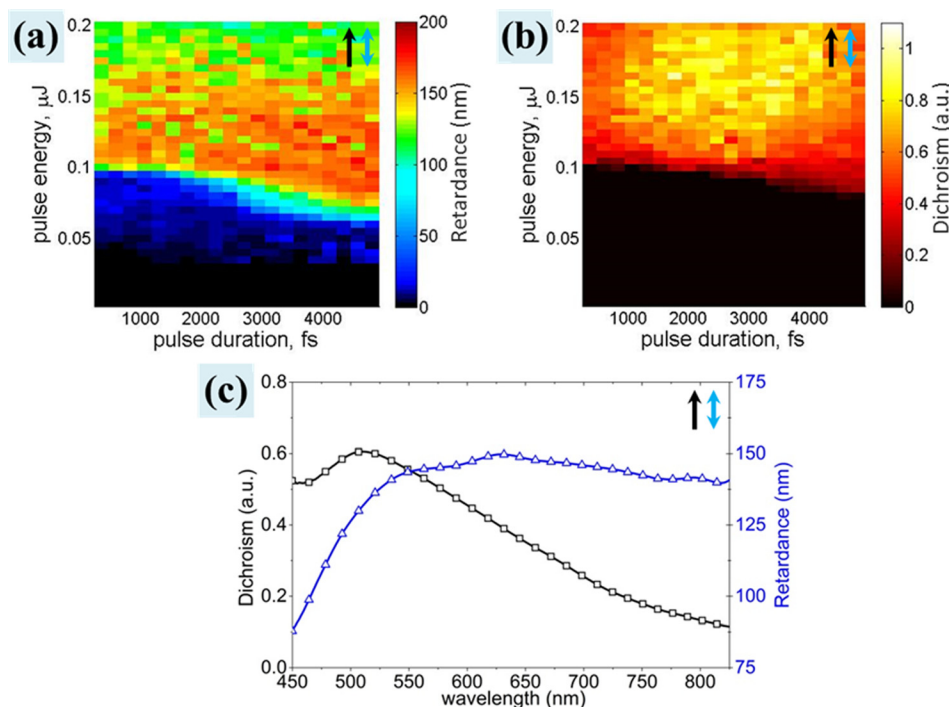


FIG. 4. Laser induced retardance (a) and dichroism (b) in a-Si:H film dependence on pulse duration and energy. The dispersion curves (c) depicted for the structure fabricated with $0.15 \mu\text{J}$ energy and 360 fs pulses show its chromatic behavior. The system was operating at 100 kHz with the writing speed of 0.2 mm/s. Black arrows indicate the laser writing direction, blue arrows—the polarization state.

For the dichroism characterization, the transmittance of light polarized parallel ($\parallel$) and perpendicular ($\perp$) to the orientation of nanograting was measured. Then the dichroism $D(\lambda)$ is defined as

$$D(\lambda) = \left| -\log\left(\frac{T_{\perp}}{T_{\parallel}}\right) \right| = \left| -\log\left(\frac{I_{\perp}I_{\parallel o}}{I_{\parallel}I_{\perp o}}\right) \right|, \quad (3)$$

where $I_{\perp, \parallel}$ and $I_{\perp o, \parallel o}$ are the light intensities transmitted through the system with the sample and without it, respectively.

The dispersion analysis performed in the spectral region from 450 nm to 825 nm for the structure fabricated with $0.15 \mu\text{J}$ energy and 360 fs pulses revealed a chromatic behavior of the laser-induced anisotropic material (Fig. 4(c)). The retardance of ~ 90 nm at 450 nm wavelength steadily increases reaching the peak value of ~ 150 nm at 550 nm, and then results in an asymptotic decrease to a plateau that corresponds well to the function of refractive index of silicon. At the same time, the extinction coefficient of silicon vanishes in the infrared and increases in the ultraviolet spectral range corresponding to direct band transitions, giving rise to stronger dichroism at the shorter wavelengths.⁵ The maximum dichroism of $D \approx 0.6$ was observed at wavelength of 510 nm, while it dropped down to $D \approx 0.1$ at 825 nm.

Both the dichroism and differential phase-shift reach the peak at 510–530 nm ($\sim 2\lambda$) wavelengths enabling the imprinting of polarizing and quarter-wave plate elements with the

applications in visible spectrum. The spectral working range of such optical elements can be tuned by the geometry (thickness and duty cycle) and material properties of the grating.^{1,41} Thus, the spectral characteristics of phase retardation and transmittance can be changed by controlling the laser processing conditions. Optical components in the range from ultraviolet to near infrared can be fabricated.^{3,5}

To demonstrate the application of laser assisted nanostructuring of a-Si:H, we imprinted Einstein's portrait (250 × 250 pixels) by encoding grayscale information via the laser beam's polarization (Fig. 5). Each $5 \mu\text{m}$ size pixel was written with the 0.2 mm/s translation speed. Rotating the linear polarization, sixteen different states in the range of 0° – 90° and 0° – 45° for the dichroic and birefringent images, respectively, were imprinted following the grayscale distribution of the portrait.

The portrait was illuminated with four polarization configurations producing the negative (parallel), positive (perpendicular), high contrast (cross-polarized), and weak contrast (unpolarised) images in optical transmission mode (Fig. 5).

In conclusion, we have demonstrated a femto- and pico-second laser assisted nanostructuring of a-Si:H film producing dichroic and birefringent material properties. Within a certain range of pulse energies, distinctive modification regimes are observed identifying hydrogen release from a-Si:H lattice (Type 0); silicon crystallization followed by the periodic surface structures self-organized parallel to the beam polarization (Type I); and silicon crystallization

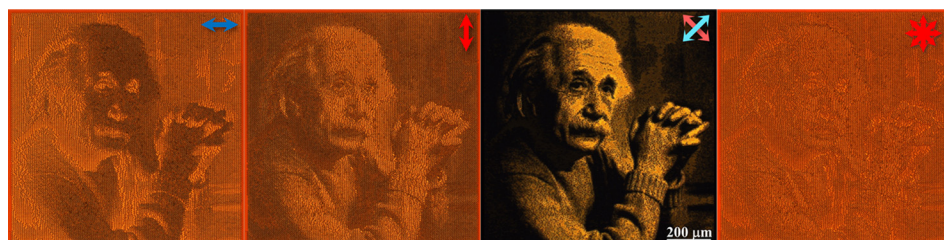


FIG. 5. The polarization sensitive Einstein's portrait imprinted in a-Si:H film by femtosecond laser nanostructuring. Arrows indicate the polarization states used for the optical transmission imaging. (Multimedia view) [URL: <http://dx.doi.org/10.1063/1.4919538.1>]

followed by the periodic structures organized perpendicular to the beam polarization (Type II). “Type I” regime defines the conditions to produce tandem modules of amorphous and nanocrystalline silicon with potential applications in photovoltaics. While “Type II” regime corresponding to the laser-induced dichroism and enhanced form birefringence is a promising alternative for fabricating functional micro-optical elements with thickness of less than 300 nm.

An anisotropic laser texturing demonstrated in this paper was implemented in the polarization sensitive ultrafast laser printing. The technique enables imprinting of optical elements with the sub-micron resolution and can be used for demonstrating ultrathin optics for complex shaping of phase and polarization, as well as in security marking or data storage applications.

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